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DEFORMATION FRACTURE AND EXPLOSIVE PROPERTIES OF
REACTIVE MATERIALS(U) CAMBRIDGE UNIV (ENGLAND)
CAVENDISH LAB J E FIELD 31 MAY 87 DAJA45-85-C-0052

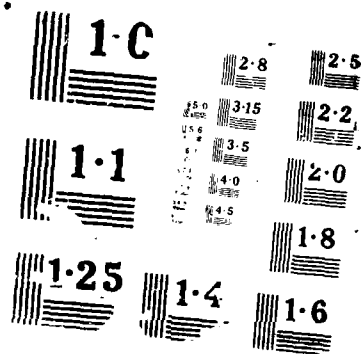
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DEFORMATION, FRACTURE AND

EXPLOSIVE PROPERTIES OF

REACTIVE MATERIALS

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Principal Investigator: Dr J.E. Field

EUROPEAN RESEARCH OFFICE

United States Army
London W1, England

Contract No. DAJA45-85-C-0052

Interim Report - 1 November to 31 December 1985

Interim report 1 Nov 85 - 31 May 87

Physics & Chemistry of Solids
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DEFORMATION, FRACTURE AND EXPLOSIVE PROPERTIES OF REACTIVE MATERIALS

1. PROGRESS

The following areas of research have either been completed or are under study since our Annual Report dated February 1985 (Contract DAJA45-84-C-0018).

(a) Mechanical properties of PBX's and their behaviour during drop weight impact

A paper on this topic was presented at the 8th Int. Detonation Symposium at Albuquerque in July 1985. It described experiments on the deformation and ignition behaviour of a range of explosives, including polymer bonded explosives (PBX's). An instrumented drop-weight apparatus was used to obtain stress-strain curves for strain rates in the range 10^2 to 10^3 s^{-1} . A novel drop-weight apparatus with transparent anvils allowed high-speed photographic recording of the impact and ignition processes. Laser speckle, used in conjunction with a specimen loaded in the Brazilian test geometry gave tensile strength and strain to failure data of PBX's. Finally, a modified Hopkinson bar apparatus gave data at strain rates up to $5 \times 10^4 \text{ s}^{-1}$.

(b) Impact experiments on thin layers of polymers and intermediate explosives

A paper on this topic was presented at the 1985 American Physical Society Conference on Shock Waves in Condensed Matter at Spokane, Washington State. It described research which extended earlier Cambridge work on the impact of explosive layers and polymers. The transparent anvil drop-weight apparatus was used to examine the effect of dynamic compression at pressures in the region of 0.5 - 1.0 GPa on PTFE and Kel-F 800 and to study the effects that these materials have upon the ignition behaviour of PETN by impact. Similar experiments have also been performed on tetryl, 3-aminotetryl and 3,5-diaminotetryl. It is shown that both polymers have a sensitising effect on PETN and this is explained in terms of their thermomechanical properties. Under impact, ignition frequently occurs with the aminotetryls but does not lead to propagation. Rapid flow precedes the ignition of 3,5-diaminotetryl but not with 3-aminotetryl. Ignition was not found with tetryl. These results are found to be consistent with previous work describing the behaviour of other energetic materials and are explained in terms of the mechanical strength properties



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and mode of failure of the explosives.

(c) Effect of physical inhomogeneities on the impact sensitivity of solid explosives: a high speed photographic study

A high-speed photographic study has been made of the initiation mechanisms during low-velocity mechanical impact of high explosives in the presence of solid glass beads and hollow glass and polymeric microspheres. In addition, a few experiments were carried out with aluminium and polycarbonate (PC) particles as additives. The explosive chosen was an oxygen-balanced mixture of triaminotrinitrobenzene (TATB) with either ammonium perchlorate (AP) or ammonium nitrate (AN). While the explosive mixture was found to be insensitive to impact, all the additives except aluminium and PC had a sensitising effect, although by different amounts. The photographic sequences revealed that initiation and propagation events are much more rapid with the glass beads compared with those with microspheres. It is believed that the 'hot spots', leading to faster decomposition rates, are generated via frictional or compressive heating mechanisms depending upon the nature of the inhomogeneity in the TATB/oxidizer/additive mixture. The results have implications for the safe-handling of explosives. The paper has appeared in Combustion Science and Technology 44, 269-278.

(d) Microstructural investigation into the fracture of PBX composites

In recent months, we have been developing polishing techniques to reveal the microstructure of PBX composites. This has allowed us to study fracture routes and to assess failure modes for some PBX's based on HMX.

(e) Automatic speckle photograph analysis for strain measurement in PBX composites

The speckle techniques described in the Albuquerque paper (1a, above) has been extended by developing techniques for automatic speckle photograph analysis and fringe processing. A paper on the topic will appear shortly.

(f) High-pressure work

A programme of research has been started to investigate the effects of high hydrostatic pressures on the optical, structural and electrical properties of explosive materials. Diamond anvil cells are used to create high pressures in small crystals which are surrounded by an organic pressurizing fluid. The optical properties, such as the band gap

edge, are monitored using a Perkin-Elmer spectrophotometer and a precision X-ray camera is used to follow the structural changes. For the elect conductivity work a Bridgman anvil apparatus is being used. So far, only single crystals of primary explosive have been examined and it is interesting to note that they can sustain hydrostatic pressures of up to ~100 kbar without explosion. We hope to obtain interesting data from further experiments.

2. RESEARCH PLANS

(a) Continued use of the techniques developed in the laboratory for studies of the behaviour of explosive samples when impacted or shocked, with particular emphasis on the processes causing 'hot spots'.

(b) A study of the effect of the particle size of HMX on its behaviour (sensitiveness, explosiveness) in the drop-weight test.

(c) Further studies on the mechanical properties of PBX's over a wide range of strain rates.

(d) Further studies of the microstructure of PBX's and their behaviour under stress, ie. failure modes, fracture paths, debonding, twinning in the explosive crystals etc.

(e) Extension of the speckle technique for recording specimen deformation to dynamic loading situations. This will involve combining laser speckle with high-speed photography.

(f) Further use of the diamond pressure cell for studies of the optical and electronic properties of some explosives.

3. CONFERENCES; PERSONNEL CHANGES

Dr J E Field, Dr M M Chaudhri, Dr J P Dear, Dr R Sundararajan and Dr C D Hutchinson attended the 8th Int. Detonation Symposium at Albuquerque. Dr Field and Dr Hutchinson presented papers.

Dr R Sundararajan attended the summer school on explosives held at Socorro.

Dr J E Field, Dr J P Dear and Dr C D Hutchinson attended the American Physical Society Conference on shock waves at Spokane, Washington State and presented 2 papers.

Dr G M Swallowe has now left Cambridge to take up a lectureship in the Physics Department at Loughborough University.

Dr C D Hutchinson who was seconded to Cambridge to study for a Ph.D. has now returned full-time to RARDE, Fort Halstead.

Dr R Sundararajan who worked in the laboratory from August 1983 to September 1985 has now returned to the INBRI Lab. in Bangalore, India.

Dr J N Johnson from Los Alamos is on sabbatical leave in Cambridge from August 1985 to July 1986.

Dr P N Howe of BRL, Aberdeen, U.S.A. is on sabbatical leave in Cambridge from September 1985 to July 1986.

Dr M Finger of Lawrence Livermore and Dr G Wakerlee of Los Alamos visited the laboratory in November 1985. Dr R Maguire of Lawrence Livermore is due to visit in February 1986

(F)

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**DEFORMATION, FRACTURE AND EXPLOSIVE
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DEFORMATION, FRACTURE AND EXPLOSIVE PROPERTIES OF REACTIVE MATERIALS

1. PROGRESS

The following areas of research have either been completed or are under study since our last Interim Report.

(a) Mechanical properties of PBX's and their behaviour during drop-weight impact

A paper on this topic was presented at the 8th International Detonation Symposium at Albuquerque in July 1985. It described experiments on the deformation and ignition behaviour of a range of explosives, including plastic bonded explosives (PBX's). An instrumented drop-weight apparatus was used to obtain stress-strain curves for strain rates in the range 10^2 to 10^3 s^{-1} . A novel drop-weight apparatus with transparent anvils allowed high-speed photographic recording of the impact and ignition processes. Laser speckle, used in conjunction with a specimen loaded in the Brazilian test geometry, gave tensile strength and strain to failure data of PBX's. Finally, a modified Hopkinson bar apparatus gave data at strain rates up to $5 \times 10^4 \text{ s}^{-1}$. This research is now being extended to the PBX's listed in Table 1, some obtained from Los Alamos and some from AWRE, Aldermaston.

Table 1

Number	Composition
Los Alamos	
PBX 9502	TATB / Kel F ; 95 / 5 (% by weight)
PBX 9501	HMX / Estane / BDNPA / BDNPF ; 95 / 2.5 / 1.25 / 1.25
X 0298	HMX / Kratan / Oil ; 97.5 / 1.125 / 1.375
X 0407	TATB / PETN / Kel F / dye ; 69.8 / 25 / 5 / 0.2
X 0344	TATB / HMX / Kel F ; 71.25 / 23.75 / 5.0
A.W.R.E.	
H	HMX / VITON ; 83.50 / 16.50
J	HMX / VITON ; 70.7 / 29.30
K	HMX / PU ; 98.0 / 2.0
P	HMX (Micronised 1000 m ² / Kg) / PU 95.0 / 5.0
R	HMX (" 500 m ² / Kg) / PU 95.0 / 5.0
S	HMX (" 300 m ² / Kg) / PU 95.0 / 5.0

(b) **Impact initiation of HMX**

The main purpose of this work was to examine, using our drop-weight machines, the effect that particle size has on the impact initiation of HMX. It has been found that coarse particles are more sensitive to impact than fine particles, although changes in the test procedure can markedly affect the relative order of sensitiveness for different particle sizes. While mechanisms such as adiabatic compression of gas spaces cannot be discounted, strong evidence was found that the prime mechanism of initiation was localised deformation in narrow bands of material by adiabatic shear. No differences in mechanical properties could be detected between discs composed of

fine and coarse HMX, but the measurement technique was suitable for detecting only bulk failure whereas the mechanical failure of the explosive disc is localized. A heat sensitive film technique was used to demonstrate the spatial distribution of hot spots which form during ignition. Lines of discolouration (which indicate heating) matched closely the failure bands which formed during impact of HMX with bare metal rollers.

For the same drop-height, coarser HMX leads to more widespread ignition than fine HMX. This was interpreted as a greater propensity for failure by shear; the higher frequency of shear bands leading to more ignition sites. Coarse HMX samples showed a faster propagation rate than fine HMX samples. Although this is thought to be largely due to shear bands providing the hot gaseous products ready access to unreacted material, the microstructure and thermal properties of the compacted disc of coarse HMX also assist this propagation.

(c) Microstructural investigation into the fracture of PBX composites

In recent months, we have been developing polishing techniques to reveal the microstructure of PBX composites. This has allowed us to study fracture routes and to assess failure modes for some PBX's based on HMX.

The research is still in progress but the following conclusions have been reached:

- (i) Polishing techniques have successfully been developed for studying two PBX compositions, one with microsecond HMX bonded with polyethylene, and the other bimodal HMX bonded with Viton.
- (ii) Earlier work has shown that HMX is susceptible to twinning (Palmer and Field 1982). Examples of twinned crystals were found in both compositions. These were presumably introduced during the handling of the HMX and the manufacture of the PBX.
- (iii) Twinned crystals were also found near the fracture origins of specimens fractured in the Brazilian test. As discussed recently (Field et al 1985), the twinning process could explain the relatively low fracture stresses of PBX's based on HMX. The point is that if the equivalent flaw size " $2c$ " is calculated for β -HMX from the Griffiths fracture criterion, taking the results given in Palmer and Field (1984) and Field et al (1985), it turns out the flaw size required for the fracture of crystals of micronised HMX is ca. 270 μm , and ca. 2.4 mm for the bimodal size distribution. This is assuming that for β -HMX, the Young's modulus, E , equals $3.10 \times 10^{10} \text{ Nm}^{-2}$ and the stress intensity factor, K_{IC} , equals $6.2 \times 10^4 \text{ Nm}^{-3/2}$ (Palmer and Field 1982). These flaw sizes are clearly much larger than the crystals! This strongly suggests that extensive crystal fracture in these composites at these stresses is unlikely, unless there are significant stress

concentrating effects. However, examination of the fracture surfaces of composition E containing micronised HMX, using scanning electron microscopy, has revealed clear examples of fractured crystals. The striations on the surfaces of the crystals are permanent twins. β -HMX undergoes elastic twinning at low stresses, which convert to permanent twins at higher stresses (Palmer and Field 1982). When a twin meets a surface, it creates a small but sharp step, which acts as a site for stress concentrations (Marsh 1960). This could explain the low stresses at which the crystals fracture.

- (iv) Several examples of crystal debonding were found. Again, this could be related to the twinning since very large interfacial stresses are set up when the twins enter a crystal and cause it to expand laterally. Since many of the twins in HMX may be elastic ones (i.e. disappear when the load reverts to zero), the deformations can be much larger than accounted for by the twins left permanently.
- (v) Several examples were found of fracture path through crystals.
- (vi) Debonding and fracture paths through crystals would expose HMX surfaces. This could well affect the explosiveness of samples.

References

- Field J E, Palmer S J P, Pope P H, Sundararajan R & Swallowe G M - Proc. 8th Int. Detonation Symp., Albuquerque (July 1985).
- Marsh D M - Phil. Mag. 5, 1197-1199, 1964.
- Palmer S J P & Field J E - Proc. Roy. Soc. Lond. A383, 399-407 (1982).

(d) Automatic speckle photograph analysis for strain measurement in PBX composites

The speckle techniques described in the Albuquerque paper (1a, above) has been extended by developing a fully automatic electro-optical system for measuring the displacement field from a double exposure speckle photograph. The optical processing section projects the power spectrum of a small region of the photograph onto a high quality image digitiser, based on a mechanically scanned photodiode array. The image is processed by microcomputer by transformation to the 2D frequency domain, where the displacement components are easily extracted. A paper has been published on this topic (Huntley 1985).

(e) Report on optical experiments on energetic materials

The optical properties of some energetic materials are being investigated using various techniques: UV-Vis transmission, infrared and Raman spectroscopy, and X-ray

diffraction. In particular the shift in the optical absorption edge has been studied as a function of temperature and pressure in order to get some understanding of how the electronic properties of these materials behave under physical stress.

Using a diamond anvil cell (DAC) the optical and phonon properties of some energetic materials are being studied, in particular silver azide, β -lead azide, PETN and RDX.

The basic design of the DAC is shown in figure 1. The sample along with a small piece of ruby is mounted in a hole approximately 0.3-0.4 mm in diameter, spark eroded in a piece of inconel alloy 0.2 mm thick. A suitable pressure medium is also required to ensure a hydrostatic pressure around the sample and for the results obtained here we used a methanol/ethanol mixture which has a working limit of over 100 kbar. The ruby is used to measure the pressure inside the gasket as the shift of its fluorescence bands with pressure is accurately known and is found to be linear over the pressure range used.

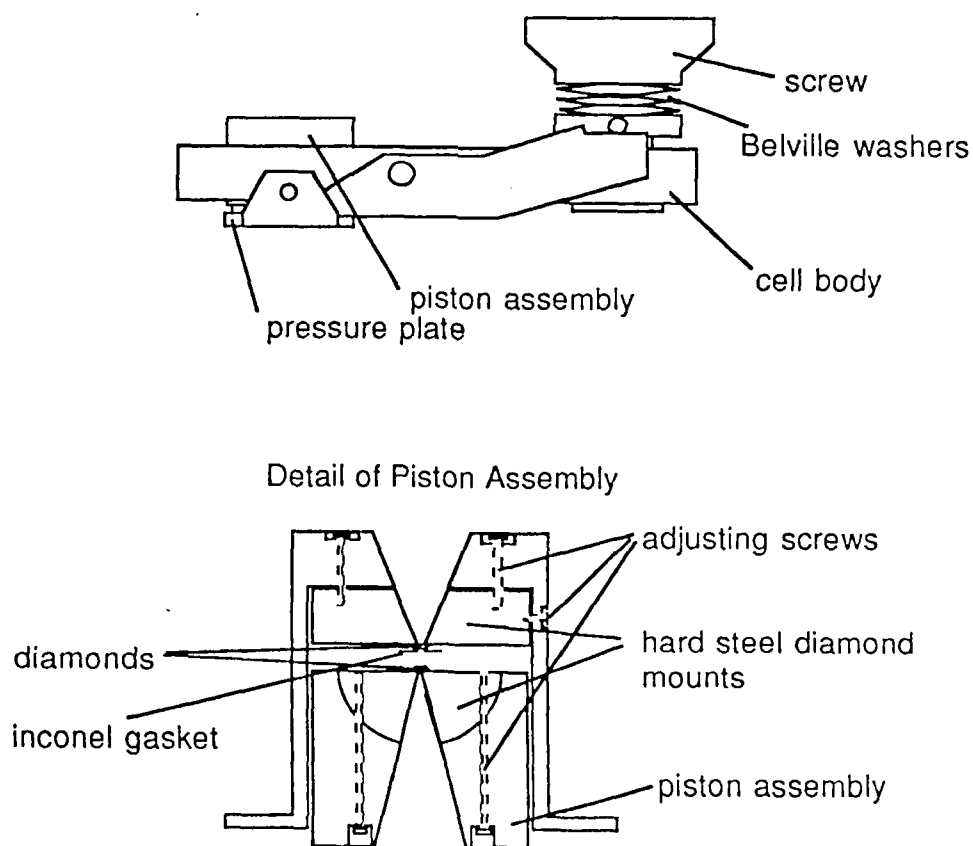


Figure 1. Basic design of the diamond anvil cell

Figure 2 shows the effect on pressure inside the DAC of tightening the pressure transmitting screw. The gasket used in this case was inconel of thickness 0.2 mm with a sample hole of 0.3 mm in diameter.

If there are any air bubbles inside the sample space, there is almost no increase in pressure until they have been collapsed (< 1 kbar). After this, the cell pressure was found to increase linearly with the number of turns of the pressure screw. In the example shown the gasket was found to distort after one complete turn so that the pressure increase was not as much as expected, and at 1.4 turns it failed completely at one edge of the diamond, releasing most of the pressure.

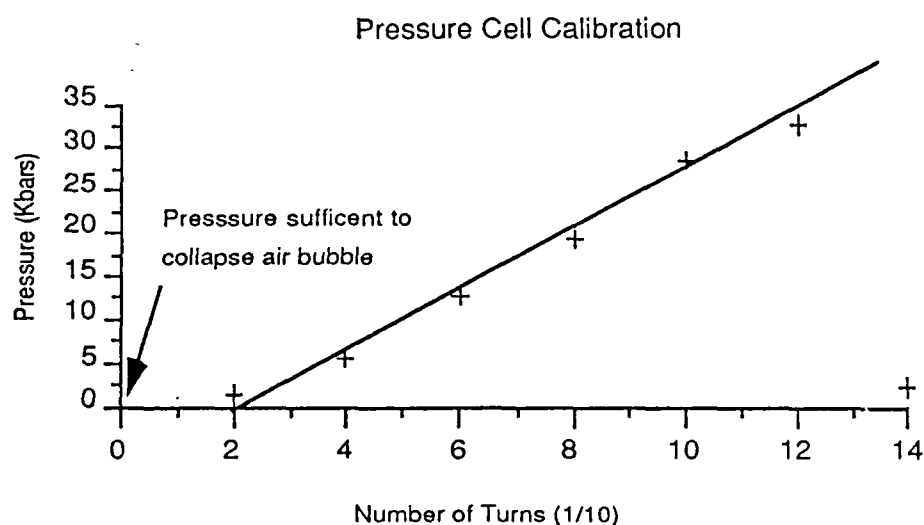


Figure 2. Effect on sample pressure when pressure screw is tightened

Results from silver azide in a mixture of ethanol/methanol (4:1) investigated under pressure with an optical microscope suggest a possible phase change at approximately 4 kbar. At this pressure the crystals are seen to change their transmission and go dark. This change is irreversible as the pressure is reduced. This darkening of the crystal causes problems with optical transmission measurements as any stray light passing between the sample and the edges of the gasket begin to 'swamp' the sample transmission as the crystal becomes dark. It is not yet known whether this darkening is due to the crystals breaking up or whether there is an actual phase change. X-ray precision measurements under pressure have not as yet shown any large change in the lattice parameters of the silver azide crystals, as they are pressurised above 10 kbar. Electrical conductivity measurements also do not

shown any phase changes below 10 kbar although a discontinuity in the electrical conductivity is seen at a higher pressure.

Measurements of the shift in band edge of β -lead azide, single crystals with hydrostatic pressure up to 30 kbar have been made. It has been found that with increasing pressure the band edge moves towards longer wavelengths (figure 3). However, the effect is quite small. Our future experiments will include other metal azides, such as KN_3 and TlN_3 .

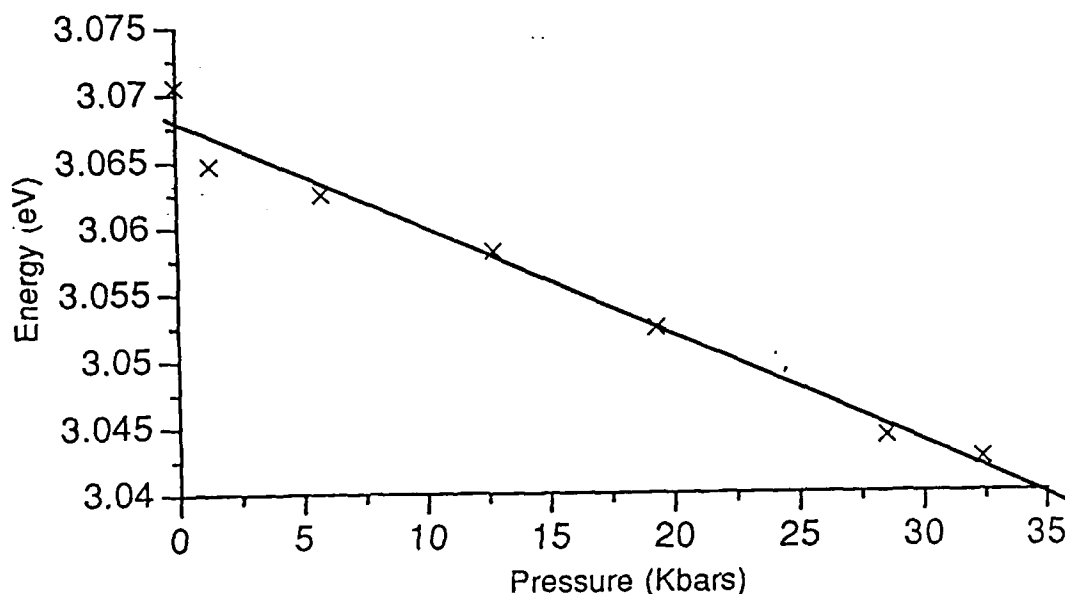


Figure 3. Shift of absorption band edge with pressure in β -lead azide

Preliminary Raman spectroscopic results for β -lead and silver azide suggest that it may be possible to follow some of the stronger Raman active phonon modes as a function of pressure in the DAC. For lead azide, the rotatory lattice modes are at 160 cm^{-1} and the strong N_3 stretching modes at around 1338 cm^{-1} (Bryant and Brooks 1971). In the second case, care is needed to avoid confusion with the strong diamond mode at 1332 cm^{-1} .

With silver azide crystals it has also been found that argon ion laser powers of less than 100 mW must be used to avoid causing the crystals to explode. This is somewhat surprising, as the wavelength used, 514.5 nm, is above the absorption edge of the crystal so that very little of the incident light should be absorbed.

2. RESEARCH PLANS

- (a) Continued use of the techniques developed in the laboratory for studies of the behaviour of explosive samples when impacted or shocked, with particular emphasis on the processes causing 'hot spots'.
- (b) Further studies of the mechanical properties of PBX's over a wide range of strain rates.
- (c) Further studies of the microstructure of PBX's and their behaviour under stress, i.e. failure modes, fracture paths, debonding, twinning in the explosive crystals, etc.
- (d) Extension of the speckle technique for recording specimen deformation to **dynamic** loading situations. This will involve combining laser speckle with high-speed photography. Further development of the automated techniques for speckle photo-analysis so that the time per data point is reduced.

3. CONFERENCES; PERSONNEL CHANGES

Dr J N Johnson from Los Alamos is on sabbatical leave in Cambridge from August 1985 to July 1986. He is doing theoretical work on the effect of changing pressure on the growth of thermal hot spots. A paper is in preparation.

Dr P N Howe of BRL, Aberdeen, U.S.A. is on sabbatical leave in Cambridge from Cambridge from September 1985 to August 1986. He is performing experimental work to classify the initiation mechanisms involved in fragment attack.

Dr J E Field's group hosted the 4th MOD (PE) Detonics Working Group Seminar on "The Application of Theoretical Studies and Modelling Techniques to the Processes of Deflagration and Detonation". It was attended by 67 delegates.

Dr J E Field has visited RARDE (Fort Halstead), RARDE (Waltham Abbey), AWRE (Aldermaston) and the Detonation Research Group at Ardeer.

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1. SUMMARY OF RESEARCH

A range of techniques has been developed over the years for studies of the behaviour of explosives when impacted and for recording their strength, failure and ignition properties. They include a drop-weight facility with transparent anvils, an instrumented drop-weight machine, a miniaturised Hopkinson bar system for high rate of strain property measurement, laser speckle for studies of deformation and fracture of PBXs, an automated system for analysing speckle and moiré records, and a heat sensitive film technique for recording the positions and temperatures of "hot spots". A paper on the heat sensitive film technique, its calibration and application for recording hot spot temperatures has been published (Swallowe, Field and Horn, 1986, J. Mater. Sci. 21, 4089).

Recent research has obtained data on the behaviour of polymer layers, binder materials, a range of PBXs, a range of HMXs of different particle sizes, and PETN when impacted in the drop-weight test. The data is in the form of force/time records and photographic observations (at microsecond framing rates). As found in earlier studies, a variety of ignition mechanisms are possible during drop-weight impact. However, a considerable amount of evidence is given both for the occurrence of localised adiabatic shear (in polymers, PBXs, HMX and PETN) and for its importance in causing ignition. Two papers are in preparation.

Significant advances have been made over the last year in understanding the drop-weight test better. Situations giving ignition during loading and during unloading have been identified and the importance of anvil deformation is shown to be important. It is shown that it is useful to describe ignition, when viewed photographically as either "local" or "widespread". A paper on this topic is in preparation.

With HMX, studies were made with samples of five different particle sizes. The coarser material gave a more violent response (i.e. had a greater explosiveness). Two important factors which influence this are: (i) the compacted structure of the impacted samples which, with the coarser material, leaves more channels (porosity) for deflagration; and (ii) the greater thermal conductivity (by a factor of 2x) of compacted coarse grain material. A paper is in preparation.

We have made significant advances in laser speckle techniques which allow the deformation and fracture of PBXs and other solids to be followed in detail. Low strain rate deformation properties of a range of PBXs have been obtained using the Brazilian test geometry for stressing samples and the automated speckle techniques for recording their deformation. Microstructural observations have been made of the failure modes in PBXs using specially developed polishing and staining techniques and microscopic observation during deformation. Papers have been published on the automated image processing system and the developed fringe analysis algorithms (Huntley 1986 J.Phys.E.: Sci. Instrum. 19, 43; Huntley 1986 App. Optics. 25, 382).

A study of Raman active phonons in both single crystal and polycrystalline samples of silver azide has been investigated in the temperature range 10K to 400K and an attempt made to correlate the phonon frequencies with the equivalent phonons in potassium azide. A paper has been submitted to Phil. Mag.

The high pressure cell and spectroscopic techniques have been used to study the effect of pressure on the band structure of silver azide and PETN in the pressure range up to 50Kbar.

Detailed laboratory reports on the above are available on request.

2. RESEARCH PLANS

- (a) Further drop-weight studies on PBXs using the transparent anvil apparatus with high speed photography, the instrumented drop-weight apparatus and the heat sensitive film technique. This combined approach is proving particularly useful for studies of the behaviour of PBXs and binder materials when imported.
- (b) Further studies of the mechanical properties of PBXs are a wide range of strain rates (up to 10^5 s^{-1}).
- (c) Further studies of the microstructure of PBXs and their behaviour under stress, i.e. failure modes, fracture paths, debonding etc.
- (d) The recently-developed automated techniques for analysing the fringe patterns from moiré and speckle records can, we feel, be speeded up significantly (a factor of about 20 times) and this we plan to do.
- (e) In the last few months, we have been able to record speckle patterns from dynamic events such as crack propagation. The successful combination of laser speckle with high-speed photography would allow deformation and crack propagation in PBXs to be studied for a range of dynamic loading situations.
- (f) High-speed framing and streak photography will be used to follow the deflagration to detonation transition of confined columns of powder explosives.
- (g) The pressure cell, with transparent anvils, designed during Dr. P. N. Howe's stay in the laboratory will be further used to film the propagation of reaction of samples following ignition.
- (h) Further Raman, absorption and X-ray studies on explosives under high pressure (up to 50Kbar) confinement.

3. CONFERENCES: PERSONNEL

- (i) Dr. J. N. Johnson from Los Alamos, USA, was on Sabbatical leave in Cambridge from August 1985 to July 1986. A paper on his theoretical work on the effect of changing pressure on the growth of thermal hot spots has now been accepted by Proc. Roy. Soc. Land.
- (ii) Dr. P. N. Howe of BRL, Aberdeen, USA, was on Sabbatical leave in Cambridge from September 1985 to August 1986. Research he started is still in progress.
- (iii) Dr. V. J. Bhasu of INBRI, Bangalore, is on Sabbatical leave in Cambridge from November 1986 to November 1987.
- (iv) Since November 1985 Dr. J. E. Field has been Chairman of the UK Explosive Materials Research Committee.
- (v) Dr. V. J. Bhasu and P. M. Dickson attended a Conference on explosives at Magréve, France, in June 1987.

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